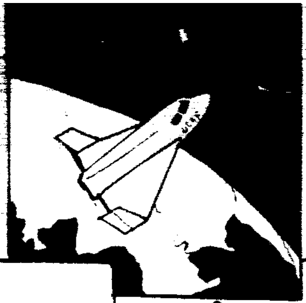

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BROMINE TRIFLUORIDE METHOD
FOR OXYGEN IN LIQUID ALKALI METALS

CONTRACT NO.: NASw-882 & CONTROL NO. 10-2379

PERIOD COVERED: (First Quarter) January 1, 1964 to March 31, 1964

AUTHORS: H Kirtchik/T Lajcik/SL Culp

Materials Development Laboratory Operation

Advanced Engine Technology Department

GENERAL ELECTRIC

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April 20, 1964

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PREPARED FOR: Chief of Materials Research Program, Code RRM, Office of Advanced Research and Technology
NASA, Washington, D. C.

TITLE: BROMINE TRIFLUORIDE METHOD FOR OXYGEN IN LIQUID ALKALI METALS

AUTHORS: Hyman Kirtchik
Thomas Lajcik
Sigmond Culp

SUBMITTED BY: Materials Development Laboratory Operation
Advanced Engine & Technology Department
General Electric Company
Evendale, Ohio

SIGNATURES:

Hyman Kirtchik, Manager
Analytical Chemistry Unit

W. H. Chang, Manager
Materials Applied Research

DATE: April 20, 1964

BROMINE TRIFLUORIDE METHOD FOR OXYGEN IN LIQUID ALKALI METALS

First Quarterly Report

January 1, 1964 to March 31, 1964

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Materials Development Laboratory Operation

ABSTRACT

33098

This program further pursues the initial successes obtained under NASr-12 wherein it was shown that the reaction of bromine trifluoride and potassium could lead to a specific method for oxygen. This requires a quantitative demonstration with superior equipment under more refined operating conditions.

The whole system has been designed with much originality, and the major portion has been constructed and assembled. It consists of: a) a helium purification section; b) a modified NASA extruder; c) a 10^{-6} Torr vacuum system; d) a BrF_3 purification and reaction system; and finally, e) a spectrophotometric oxygen measuring section.

Calibration, leak checking, and further initial tests are in progress.

BROMINE TRIFLUORIDE METHOD FOR OXYGEN IN ALKALI METALS

TECHNICAL BACKGROUND

The research on NASr-12 program[1] over the past years had as its objective the development of a method for the determination of oxygen in alkali metals with both precision and accuracy in the lower ranges. A 1963 survey of all methods for oxygen in all alkali metals points to the wisdom of the original premise by NASA that such a need must still be fulfilled. Even today, the application of potassium, for example, for use in nuclear turbomachinery is limited by the quality of the alkali metal.

The real effects of corrosivity by potassium cannot be established until the accurate level of impurities, particularly oxygen, is known with reasonable certainty. Towards this direction, a great deal of time had been spent on the development of a method of sampling that would have universal appeal and application to the preferred methods of any laboratory. This, plus a number of minor developments on automatic titration, alkali metal transfer, etc., have been widely adopted.

Before it is safe to conclude that certain materials of containment at elevated temperature can stand any alkali metal, it will be necessary for different laboratories to agree on the values for oxygen. Unfortunately, it is possible at this point in time for more than one laboratory to agree on this "oxygen value" while both have different oxygen values! This comes about because other contaminants within the alkali metal are unavoidably finally called oxygen in all three of the present analytical methods in vogue. In an attempt to circumvent all of the difficulties attendant with present state-of-the-art methods, the research here (1962-1963) was directed at an original hydrogen fluoride method which had as its main objective specificity for oxygen.

The development activities uncovered certain difficulties with the $HF-F_2$ approach. The direction of the program was changed to reactions involving bromine trifluoride.

[1] "Research on Analytical Methods for Oxygen in Liquid Alkali Metals", H. Kirtchik and T. Lajcik, DM 63-233, General Electric Company, September 30, 1963.

The previous study conclusively established the feasibility of developing a specific method for the analysis of traces of oxygen (5 to 150 ppm) in alkali metals. The method is based on the reaction of purified bromine trifluoride (BrF_3) with the subject metal (M) to liberate molecular oxygen which is then dynamically determined by the Brady method.

$$6MO + 4BrF_3 = 6MF_2 + 2Br_2 + 3O_2$$

When reacted with bromine trifluoride, excellent recoveries of oxygen were obtained on test samples of potassium bromate ($KBrO_3$) containing between 25 and 150 micrograms of oxygen. Thus, the BrF_3 method is applicable for analytical purposes at concentration levels as low as three orders of magnitude less than previously reported in the literature.

The NASr-12 program also demonstrated qualitatively the feasibility of applying the method to potassium by the reaction of BrF_3 with a contaminated potassium sample, which resulted in a large measurable recovery of oxygen. The tests also showed that BrF_3 can be made under specific conditions to react in a controllable manner with potassium metal without observable difficulties.

The NASr-12 program was then concluded by showing that BrF_3 could lead to a novel and referee quality chemical method. This would require a demonstration with superior equipment under more refined operating conditions. This new program from NASA now allows for this demonstration, and the plan of attack follows the work statement contained therein.

PLAN OF ATTACK

To determine the quantitative aspects of this reaction, a refined apparatus is being constructed with the awareness of the individual highly reactive nature of both the alkali metal (K) and the interhalogen (BrF_3) and their containment. Secondly, the apparatus must exclude air and oxygen, the very element whose adventitious presence must be rigorously avoided if one is to obtain meaningful and sensitive results.

The development program is subdivided into three phases:

I The design and construction of the over-all system.

II Calibration of sections of the system.

III Determinations of oxygen in potassium under varying conditions using statistical methods and with purposeful additions of oxygen-bearing compounds.

EXPERIMENTAL (PHASE I)

The complete system is described below, including the status of each section.

1. The test apparatus has been designed and nearly completely constructed to incorporate the following features:

   a) A storage, purification, and transfer system for bromine trifluoride. In the previous NASr-12 work (Page 7),[1] bromine trifluoride from an upright cylinder was drawn into an evacuated and frozen Kel-F trap. Hot water in a container around the cylinder aided in the transfer. The whole process was laborious and required several hours before even 50 ml of the reagent were carried over. The liquified BrF_3 was then forced over into the Reaction Cell wherein purification was then effected just prior to a run. This consumed further time. To circumvent these difficulties, the new system (see Figure 1) has the cylinder inverted and then connected to a large 500-650 ml Monel Cylinder (as in Figure 2). By simple evacuation with a roughing pump, it is expected that large amounts of BrF_3 will freely transfer to the monel container within a shorter period of time. The purification of the BrF_3 will take place here and in advance of need. The container is constructed identically to those in Drawing #4012093-787. A mating flange is being made with several pipe leads for BrF_3, helium, and evacuation.

   Previous experience indicated that purification was obtained by simple refluxing or pumping at room temperature. The alternate addition of helium and freezing resulted in the absorption of much of this gas. On

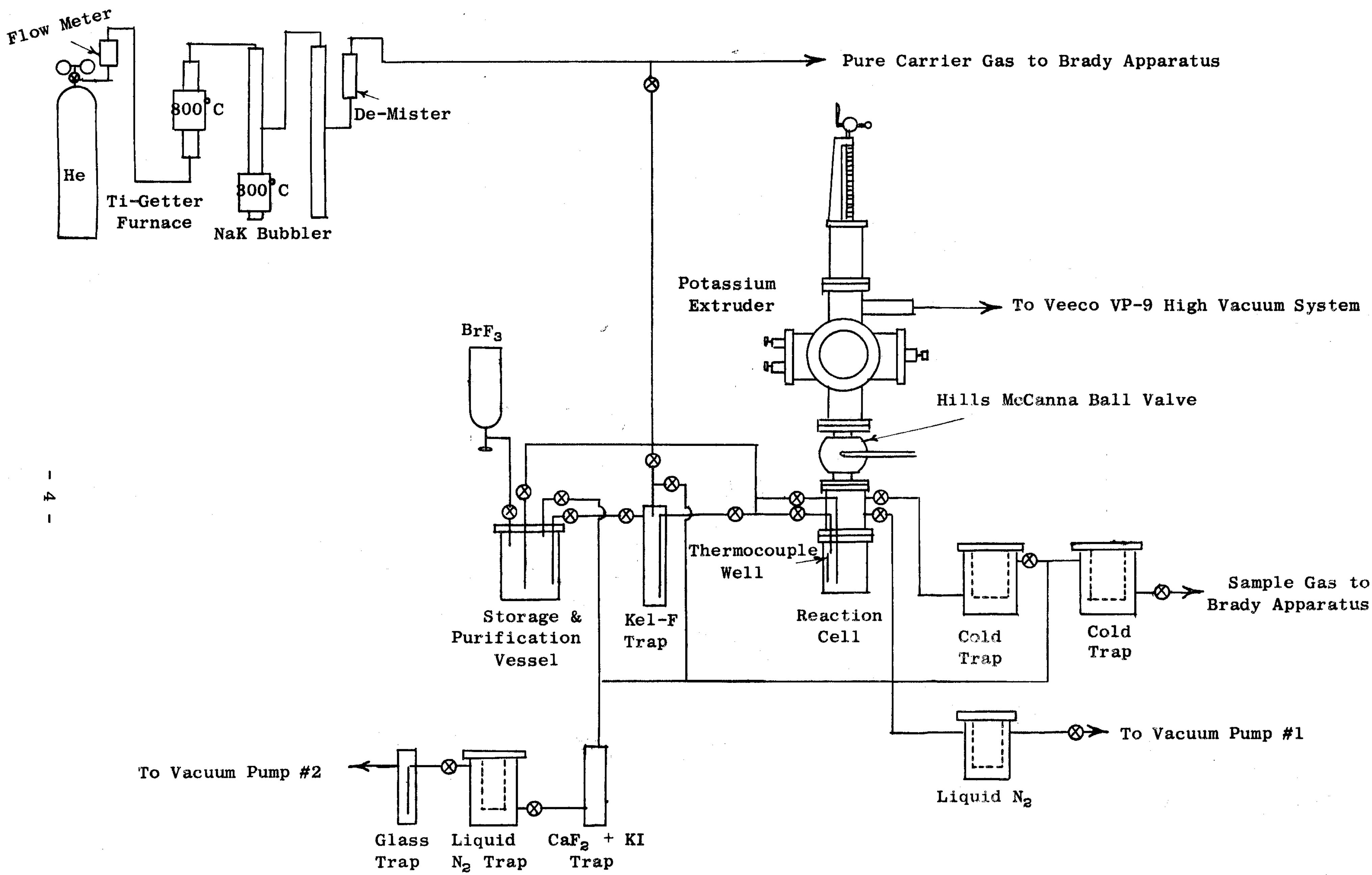

FIGURE 1 Over-all System Design for BrF_3 Determination of Oxygen in Alkali Metals

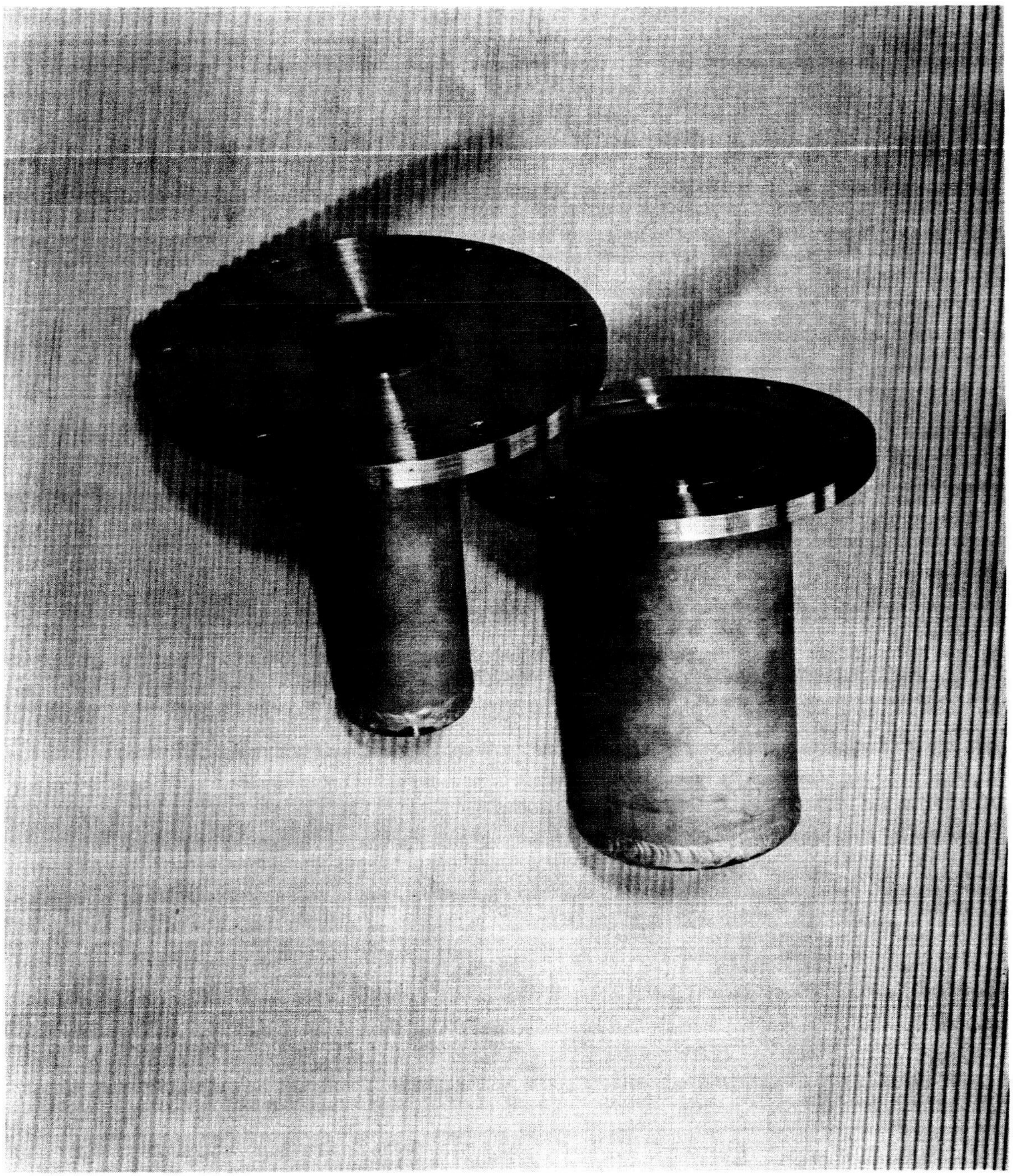

FIGURE 2 Construction of Monel Traps and Cylinders

C 4012093-787 REV 1

ITEM	IDENTIFICATION NO.	DESCRIPTION OR NAME	ZONE	GROUP NO. & QTY 1	2	3	4
1		ASSY	A6	X			
2	4012093-787P2	PLATE	B4	1			
3	P3	PIPE	B4	1			
4	P4	FLANGE	C4	1			

REVISIONS

SYM	ZONE	DESCRIPTION	DATE	APPROVED
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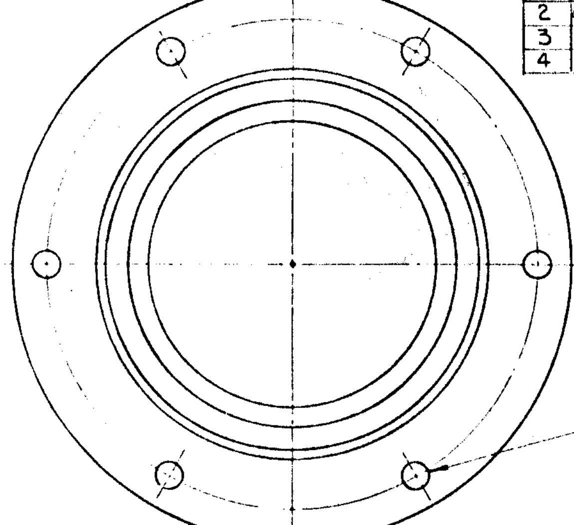
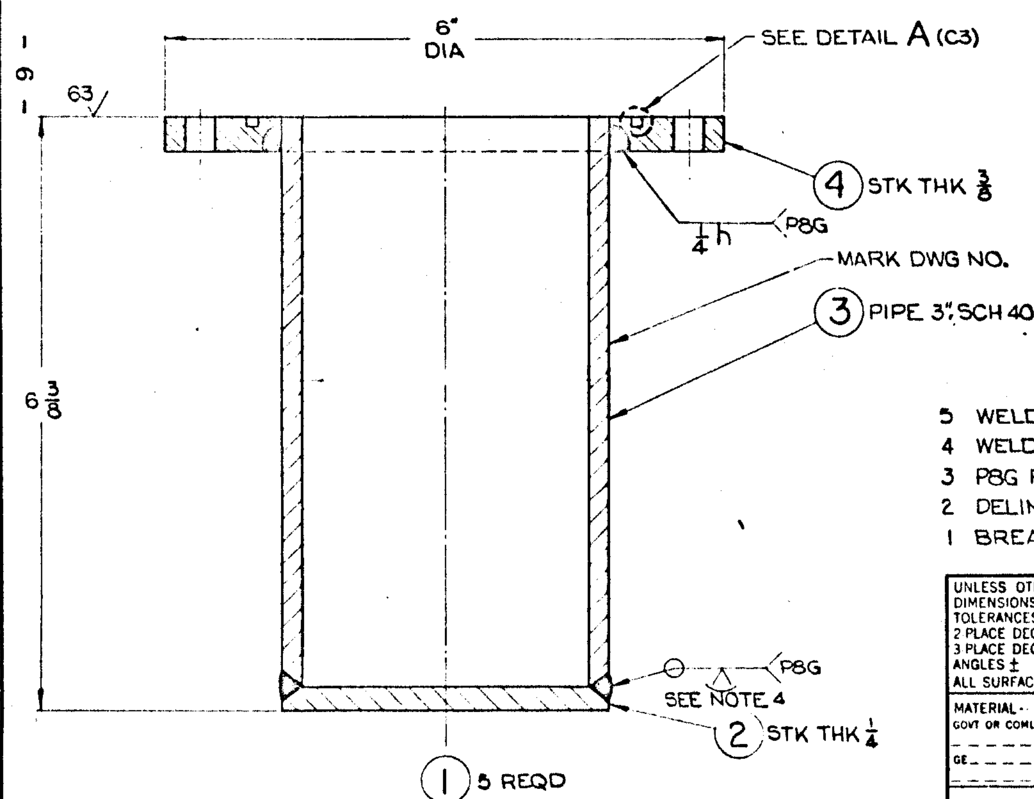
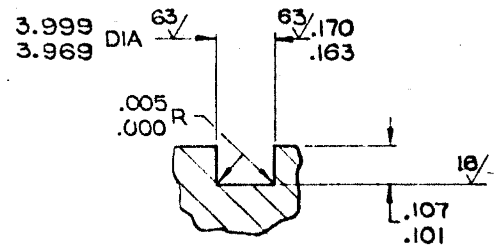






DETAIL A (C5)
SCALE ~ 4/1

5 WELDS MUST WITHSTAND 10^{-5} - 10^{-6} TORR WITH NO LEAKS
4 WELD TO BE FULL PENETRATION
3 P8G FILLER MATL: INCO 60
2 DELINEATED RIGHT ANGLES 90° ± 30' UNLESS OTHERWISE SPECIFIED
1 BREAK SHARP CORNERS .015 MAX RAD OR CHAM

REDUCED MICRO-COPY

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES — TOLERANCES ON FRACTIONS ± 1/32
2 PLACE DECIMALS ±
3 PLACE DECIMALS ±
ANGLES ±
ALL SURFACES 125

MATERIAL - GOVT OR COML MONEL 400 SERIES

GE

SIGNATURES	DATE
C.H. Kennedy	2/19/64
C.H. Kennedy	2/20/64
ISSUED FR Uester	2-21-64
ENGRG [illegible]	2-20-64
MFG K.A. Eveleth	2/20/64
MATLS H. Kirchek	2-20-64

GENERAL ELECTRIC
AET DEPT LOC EVENDALE PLANT

TITLE
REACTION CELL OR TRAP

CONTRACT NO.

CODE IDENT NO	SIZE	DWG NO.
06083	C	4012093-787

SCALE 1/1 WT SHEET OF 57

AR. 394

FN-903-P (3-63) PRINTED IN U.S.A.

C 4012093-787 REV 1

thawing and further pumping the helium aided in the removal of the impurities common to bromine trifluoride.

	m.p. °C	b.p. °C
BrF_5	-61.3	40
OF_2	-223.8	-144.8
HF	-92.3	19.4
Br_2	-7.2	58.8
BrF_3	8.8	135

In addition to the easy removal of the first four high vapor pressure gases, metallic fluorides remain behind or are inert later on. With the container materials used in the cylinder (ferritic) and in the monel cylinder (Ni-Cu), no oxyfluorides are present.

Exact amounts of liquid BrF_3 can then be transferred to the Kel-F trap between the Monel Cylinder storage and the reaction vessel. As before, this small flared Kel-F cylinder with its monel head is a direct copy of that used at both Argonne and Oak Ridge National Laboratories. It will be calibrated for its volume content. Within a short space of time, it is expected that the BrF_3 forced into it from the monel storage cylinder by helium can then be quickly forced into the reaction cell. After this, additional BrF_3 can be added to the Kel-F trap and forced over, etc., until a known amount of the bromine trifluoride is then contained within the reaction cell.

The storage cylinder is nearly completed and essentially consists of 3" monel pipe heliarc welded to sheet monel. The upper section has been designed as a flange with "O" ring grooving made with another monel flange. Suitable fittings attached to drilled holes in this latter flange will allow for the entrance and exit of the BrF_3, evacuation directly to the catch-all traps, and for helium purging. The "O" rings used here and elsewhere will be either Viton or Teflon.

b) A main reaction cell for the accommodation of a large volume of bromine trifluoride to allow for the sequential determination of multiplicate alkali metal samples.

The main Reaction Cell has been enlarged to contain over 500 ml of liquid bromine trifluoride. The design of the cell is shown in Drawing #4012093-787. It is made of 3" monel pipe and sheet stock. This design allows for the introduction of a large number of sequential samples of alkali metal. Figure 2 shows the cell (this is identical to the outer trap member shown).

The reaction cell is connected to the exit end of the large ball valve by an adaptor as shown in Drawing #4012093-785. This is also constructed of sheet monel and 3" pipe. Several holes (not shown) will be drilled into the pipe to allow for the entrance of helium, BrF_3 from the Kel-F trap, and for evacuation. $\frac{1}{4}$ or $\frac{3}{8}$" tubings will be attached and descend into the Reaction Cell. Figure 3 shows the Adaptor.

c) Lines of communication between valves, helium pressure system, evacuation system, and traps for the proper conduction of oxygen and helium to the measuring system. Precision grade valves, joints, and seals of corrosion resistant materials shall be used to insure system integrity.

The connections between all members will be made with monel tubing and pipe fittings with welded construction. In some instances where pipe threads are involved, Teflon tape will be used in conjunction with them. There has been difficulty in obtaining all monel valves with Teflon seals and straight flow-through construction. The seals on all mating flanges are "O" rings of Viton and Teflon.

The lines for helium are made of copper tubing and Swagelok fittings.

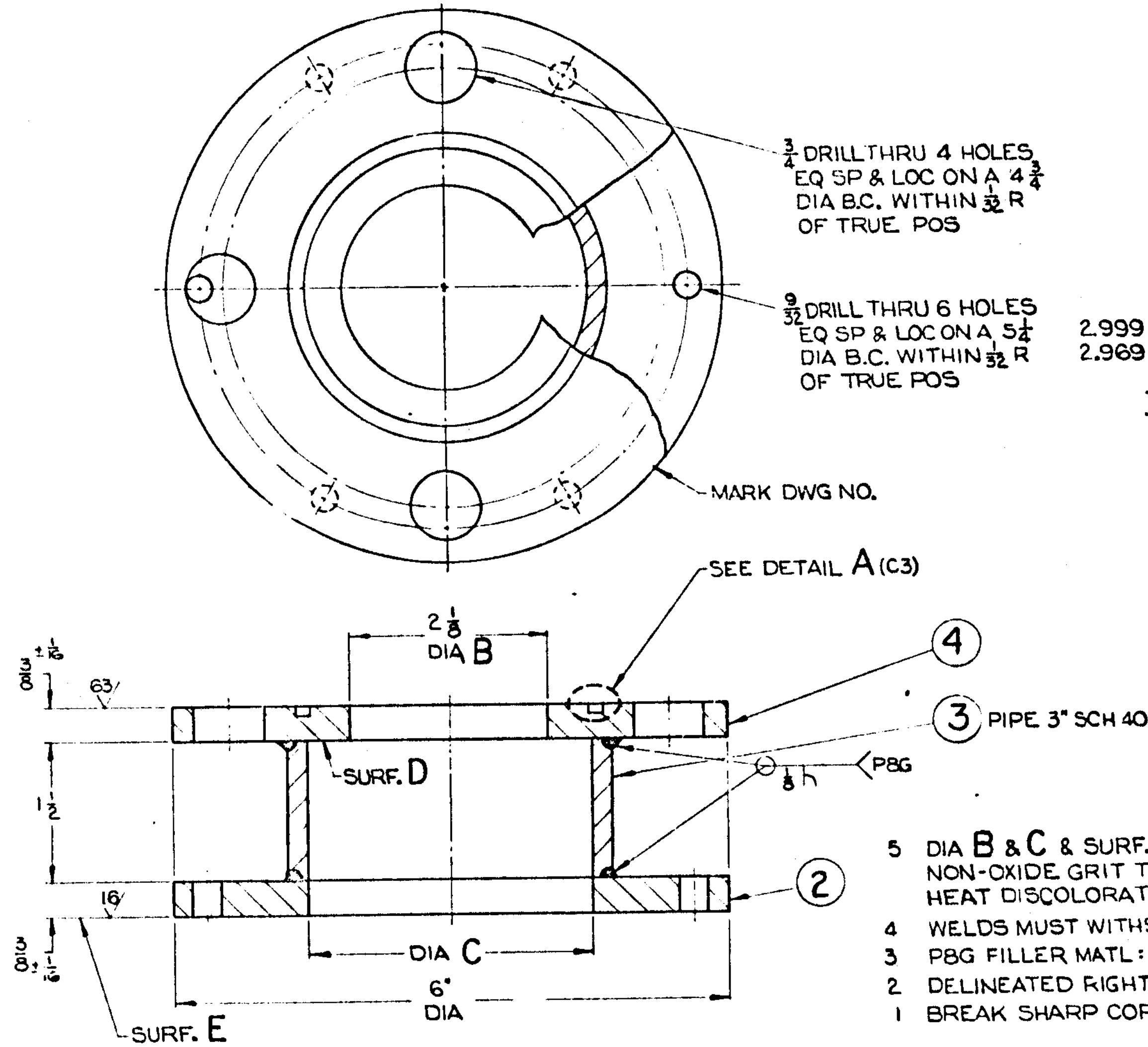
The monel traps have been designed to avoid clogging or otherwise impeding the flow of helium plus oxygen. Drawing #4012093-786 shows the inner member of a trap. This allows for top loading of liquid nitrogen. This Inner Trap Member mates with the outer member shown in Drawing #4012093-787. Figure 4 shows the manner of assembly. Holes will be drilled into the outer 3" trap for intercommunication with other sections of the apparatus by the aforementioned pipe or tubing.

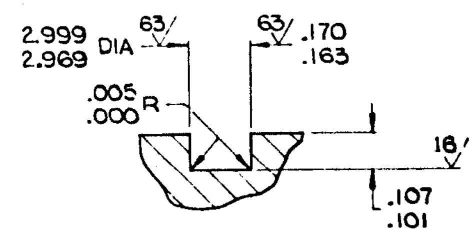
C 4012093-785 REV 1

ITEM	IDENTIFICATION NO.	DESCRIPTION OR NAME	ZONE	GROUP NO. & QTY 1	2	3	4
1		ASSY	A6	X			
2	4012093-785P2	FLANGE	B4	1			
3	P3	PIPE	C4	1			
4	P4	FLANGE	C4	1			

REVISIONS

SYM	ZONE	DESCRIPTION	DATE	APPROVED
1		GENERAL CHANGES AR-394	FEB. 24, 1964	C.I. Kennedy





DETAIL A (C5)
SCALE ~ 4/1

5 DIA B & C & SURF. D & E MUST BE VAPOR BLASTED WITH NON-OXIDE GRIT TO REMOVE VISIBLE OXIDES, SLAG & HEAT DISCOLORATION FROM WELDING
4 WELDS MUST WITHSTAND 10^{-5}-10^{-6} TORR WITH NO LEAKS
3 PBG FILLER MATL: INCO 60
2 DELINEATED RIGHT ANGLES 90° ± 30' UNLESS OTHERWISE SPECIFIED
1 BREAK SHARP CORNERS .015 MAX RAD OR CHAM

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES — TOLERANCES ON: FRACTIONS ± 1/32
2 PLACE DECIMALS ±
3 PLACE DECIMALS ±
ANGLES ±
ALL SURFACES 125

MATERIAL — GOVT OR COML MONEL 400 SERIES

SIGNATURES	DATE
C.I. Kennedy	2/19/64
C.I. Kennedy	2/20/64
F.R. Vester	2-21-64
W.R.T.L.	2-20-64
R.A. Roberts	2/20/64
N. Kirchner /Frv	

GENERAL ELECTRIC
AET DEPT LOC EVENDALE PLANT

TITLE REACTION CELL - BALL VALVE ADAPTER

CONTRACT NO.

CODE IDENT NO	SIZE	DWG NO
06083	C	4012093-785

SCALE 1/1 WT SHEET OF 57

AR. 394

C 4012093-785 REV 1

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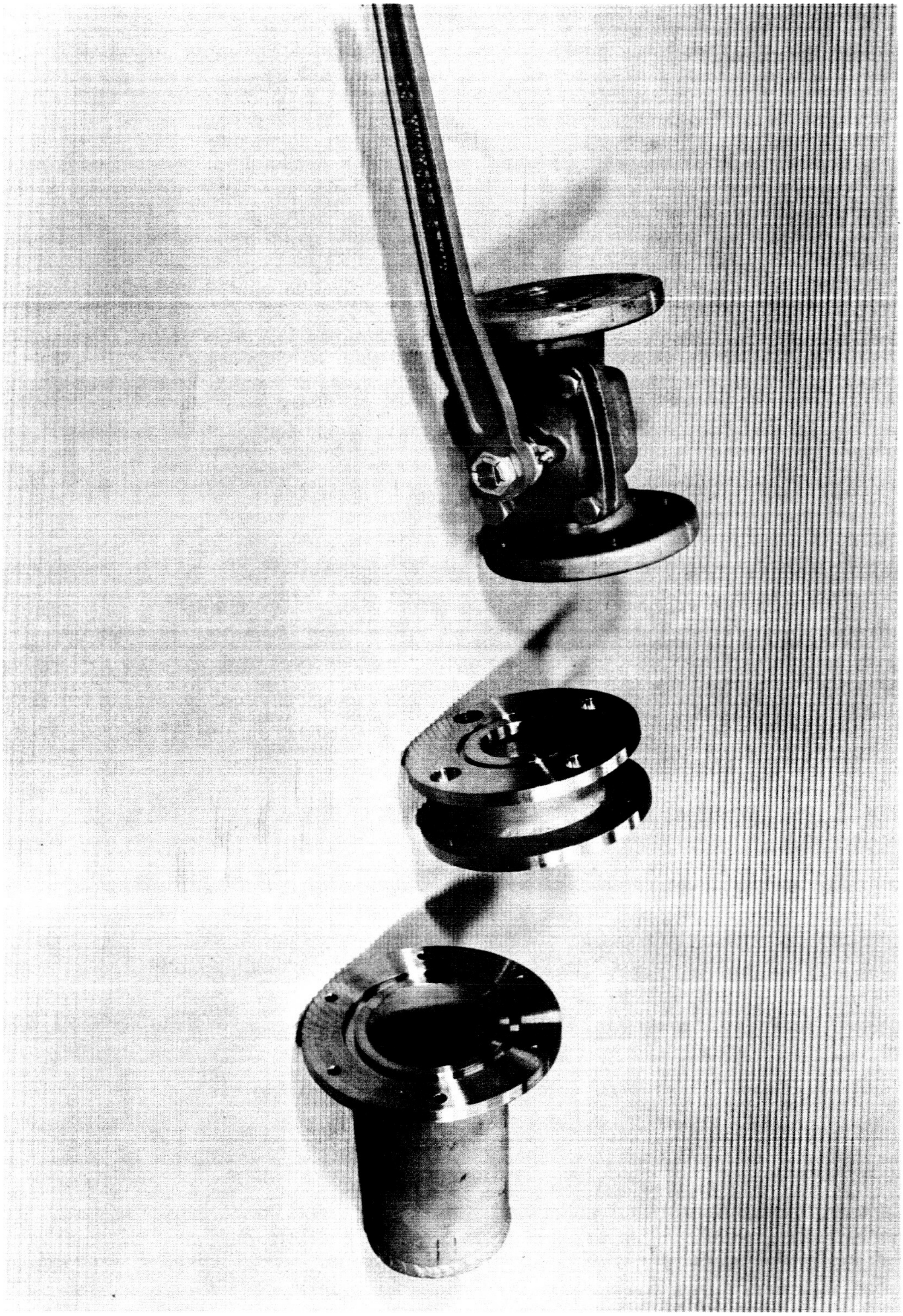
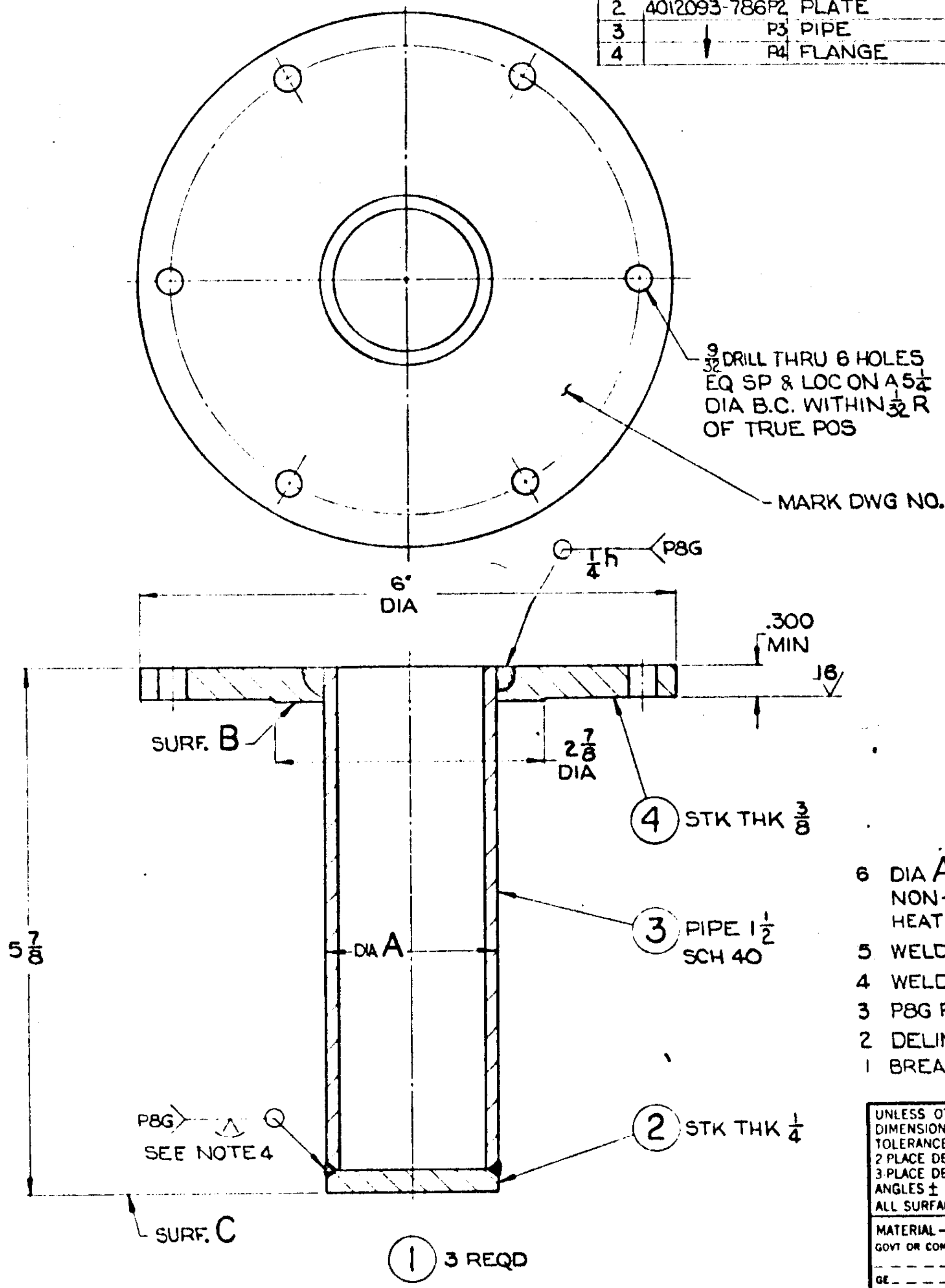


FIGURE 3 Monel Ball Valve-Adaptor-Reaction Cell Combination

ITEM	IDENTIFICATION NO.	DESCRIPTION OR NAME	ZONE	GROUP NO. & QTY 1	2	3	4
1		ASSY	A6	X			
2	4012093-786P2	PLATE	A5	1			
3	P3	PIPE	B5	1			
4	P4	FLANGE	B5	1			

REVISIONS				
SYM	ZONE	DESCRIPTION	DATE	APPROVED
1		GENERAL CHANGES AR-394	FEB. 24, 1964	C. H. Kennedy



6 DIA A & SURF. B & C MUST BE VAPOR BLASTED WITH NON-OXIDE GRIT TO REMOVE VISIBLE OXIDES, SLAG & HEAT DISCOLORATION FROM WELDING
5 WELDS MUST WITHSTAND 10^{-5}-10^{-6} TORR WITH NO LEAKS
4 WELD TO BE FULL PENETRATION
3 P8G FILLER MATL: INCO 60
2 DELINEATED RIGHT ANGLES 90° ± 30' UNLESS OTHERWISE SPECIFIED
1 BREAK SHARP CORNERS .015 MAX RAD OR CHAM

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES - TOLERANCES ON FRACTIONS ± $\frac{1}{32}$
2 PLACE DECIMALS ±
3 PLACE DECIMALS ±
ANGLES ±
ALL SURFACES 125√

MATERIAL - GOVT OR COML MONEL 400 SERIES
GE

SIGNATURES	DATE
C. H. Kennedy	2/19/64
C. H. Kennedy	2/20/64
F.R. Vester	2-21-64
[illegible]	2-20-64
K.A. Collett	2/20/64
H. Kirchner /prev	

GENERAL ELECTRIC
AET DEPT LOC EVENDALE PLANT

TITLE INNER TRAP MEMBER

CONTRACT NO.
CODE IDENT NO 06083 | SIZE C | DWG NO. 4012093-786
SCALE 1/1 | WT | SHEET OF

57

AR-394

C 4012093-786 REV 1

REDUCED MICRO-COPY

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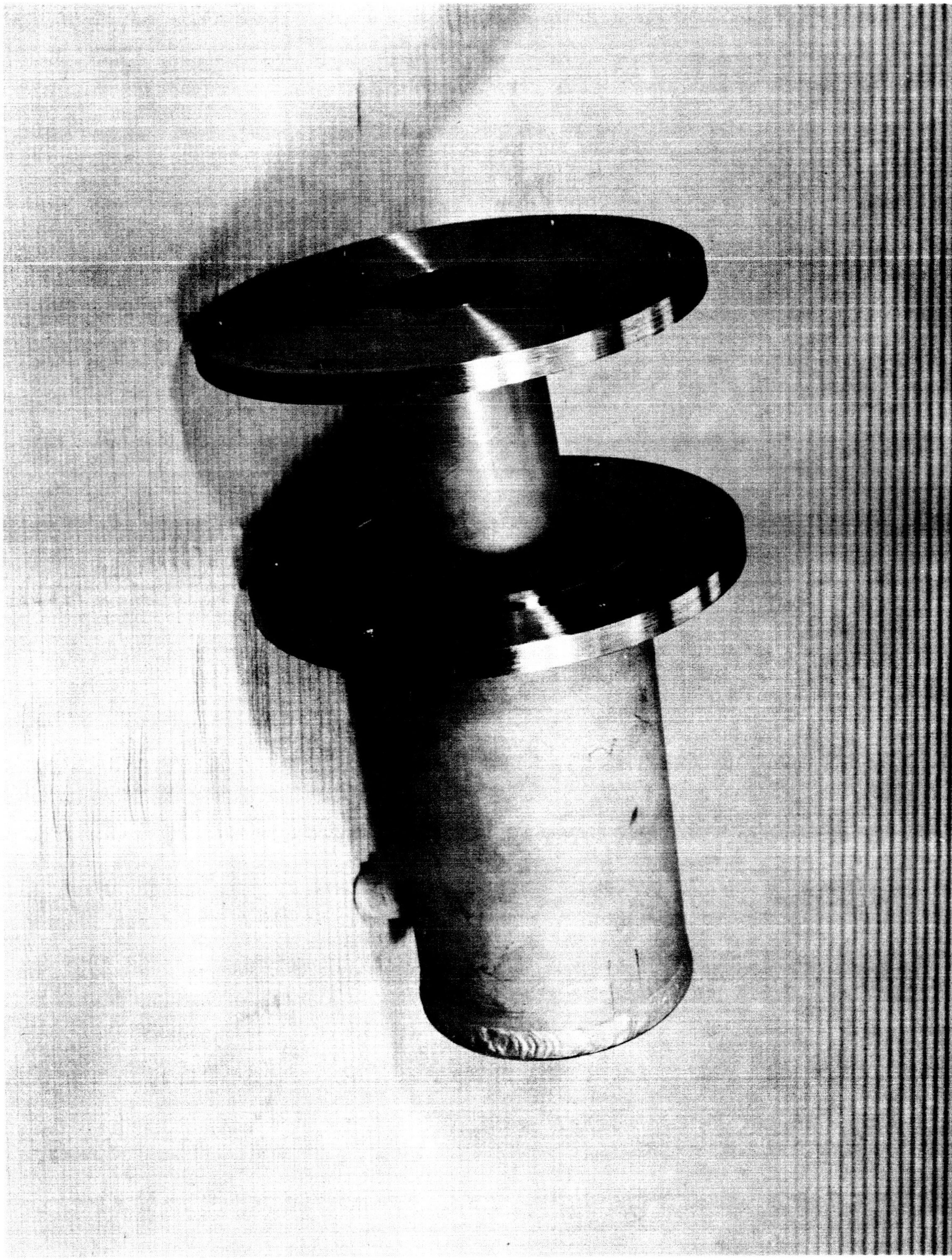

FIGURE 4 Monel Cold Trap Assembly

d) An extrusion system similar to that designed by NASA. Several modifications have been made to tie into the over-all system. Since it has been recently demonstrated by NASA that oxygen values one can expect (by the Amalgamation Method) are related to the degree of vacuum <u>during</u> extrusion, a more rigorous control has been designed. A change in the method of operating the actuators to cut the potassium metal and to catch waste alkali metal has been made. An end plate of the extruder is shown in Figure 5. Veeco bellows vacuum valves with the side port of each sealed are used to provide the lateral motion for the cutters and drop-off tray. The end of this bellows will be sealed to the rods that guide the cutting wire. This arrangement will allow for better maintenance of vacuum. The improvement is that a static "O" ring type seal is used rather than one depending on friction (High Vacuum Shaft Seal from Vacuum Research Corp.) between the cutter rod and encircling rubber washers to make a tight seal during operation. The approximately $1\frac{1}{4}$" travel of the bellows is sufficient for the cut-off procedure.

The top seal of the extruder, which accommodates the $\frac{1}{4}$" plunger rod is not easily modified by a bellows arrangement. However, to overcome the usual rubber washer seal, a unique design and construction has been made. As noted in Figure 6, two standard brass Consolidated Vacuum Corp. double "O" ring sliding seals (Type SR-25) were mounted one on top of the other by means of a hollow copper tubing. A port from the hollow tube by another smaller sized tubing allows for evacuation between the seals during extrusion. The seal, therefore, consists of four "O" rings with a pumped out volume between them.

The rear sight-glass window (behind the tube of potassium) has been extended out somewhat to allow for a two-inch tubing attached at right angles to it. A flange on this extra tubing connects to the vacuum system described later on. This allows for evacuation of the whole extruder while closed off and isolated from the reaction cell.

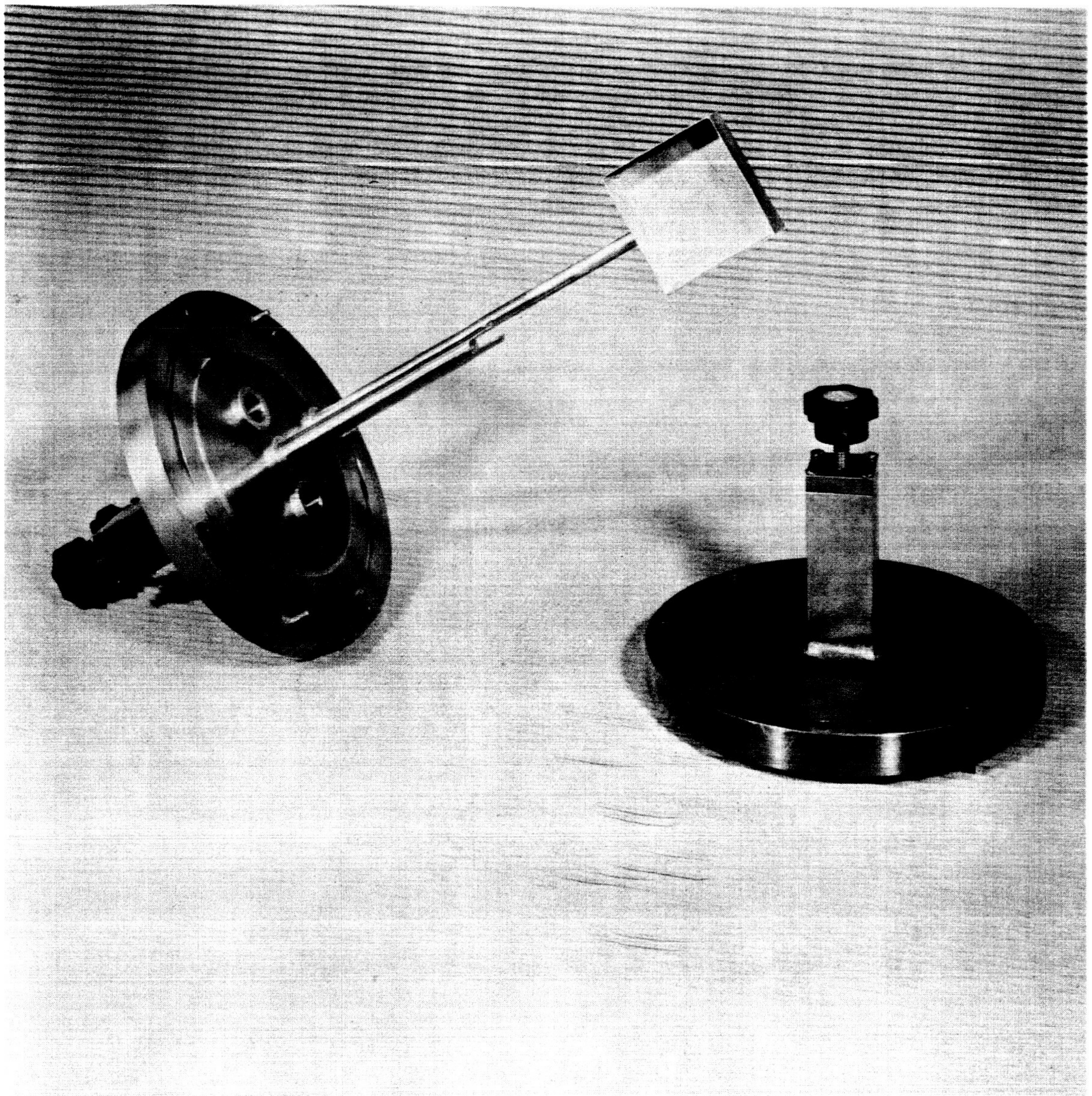

FIGURE 5 Extruder End-Plates with Bellows-Sealed Activator Rods

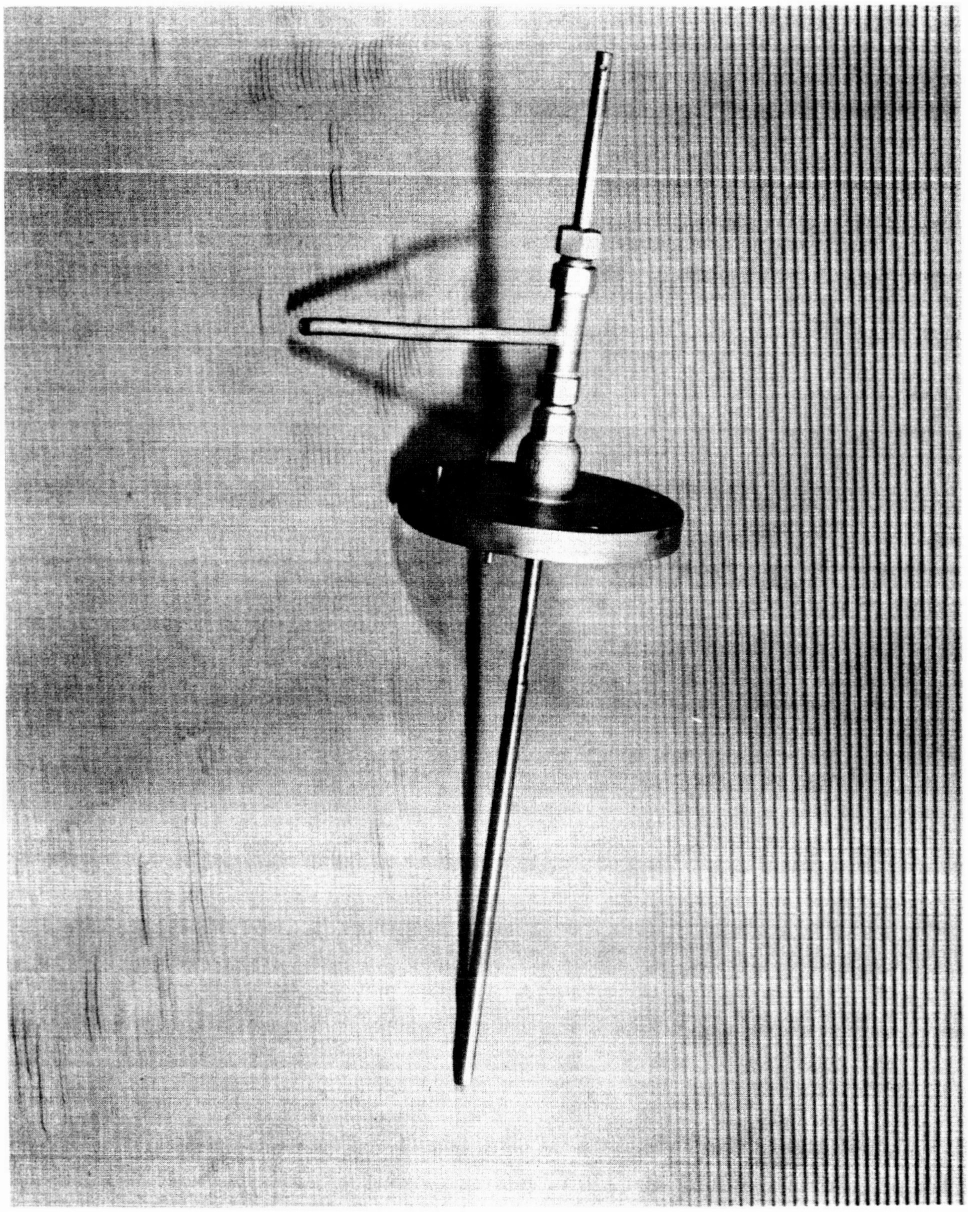

FIGURE 6 Double Vacuum Feed-Through with Extruder-Rod

The flange on the exit lower end of the extruder has also been changed to mate with a ball valve which, in turn, will connect to the Reaction Cell. It is a Hills McCanna "McCannaseal" top entry 2" all monel ball-valve with flanged ends. Teflon packing and "O" rings are used exclusively. The valve has been tested to 10^{-6} Torr. As noted previously in Figure 3, the ball valve is shown with the Adaptor and the Reaction Cell in the manner in which they will be joined by "O" ring seals.

e) A vacuum system to evacuate the over-all system and with appropriate baffles and traps to minimize system contamination. The capability must be between 10^{-5} and 10^{-6} Torr.

High vacuum in the extruder and reaction cell is obtained by means of a Veeco VP-9 pumping system connected to the extruder by means of the two inch port and line. The VP-9 system consists of three basic elements: (1) an EP-2AB three stage fractionating diffusion pump; (2) a "low loss" liquid nitrogen cold trap; and, (3) a Welch 1402 VEB mechanical fore pump. These basic components, along with attendant plumbing, valves, and gauges comprise a system capable of attaining an ultimate vacuum of less than 10^{-6} Torr.

In order to further minimize back-streaming, the system is provided with a water-cooled diffusion pump in place of the customary air-cooled model whose back-streaming rate is about .003 cc per hour at 25°C. The pumping speed is about 85 liters per second.

Back-streaming is further reduced by a liquid nitrogen cold trap of 0.8 liter capacity. The cold trap, of stainless steel and kovar construction, will function at liquid nitrogen temperature for 6-8 hours per filling.

The 5 CFM fore pump can also be used for rough-pumping of the reaction cell by means of an auxilliary vent valve of the quick-acting toggle type.

System pressures will be monitored by means of two thermocouple gauges and a Bayard-Alpert type RG-75-KF ionization gauge located at the liquid nitrogen trap inlet. One of the thermocouple gauges is situated at the inlet port while the other will be used to measure foreline pressures.

A $^3/_4$" quick-coupling is also provided in the system for installation of a mass spectrometer leak detector or a backfill gas cylinder.

f) A helium NaK purification system and helium flow control system.

Linde commercial tank grade helium is metered into the system by a Matheson Model #8-AF-590 two stage regulator with flow meter. Initial purification is accomplished by a titanium getter furnace operating at about 800°C. This furnace consists simply of an Inconel tube approximately 26 inches long and $2^1/_2$ inches in diameter. The tube is filled with titanium sponge and the central portion of the tube is heated by a Marshal tube furnace.

Final purification of the helium is achieved by passing the gas through a Mine Safety Appliances Corp. Model 15 NaK bubbler. See Figure 7. Upon entering the bubbler the gas first passes through a "hot leg" containing about $^1/_3$ pound of NaK (44-56% K) at 300°C. It then passes through a "cold leg" containing the same amount of NaK at room temperature, and then through a demister to remove entrained metal vapor. Flow rates up to 1 SCFM at pressures as high as 150 psig can be maintained without entrainment. Operational life without recharging is approximately 3,000 hours. The gas stream, exiting from the bubbler, is split to provide both the Brady apparatus and the analytical system with pure helium.

2. The oxygen measuring system has been designed and constructed and consists of:

a) All glass modified system as in Figure 8 incorporating the Brady principle and sensitive to 0.1 to 100 μg of oxygen per hour. In essence, oxygen (in the helium stream) quantitatively reacts with sodium anthraquinone-β-sulfonate to change an alkaline solution of the latter from red to

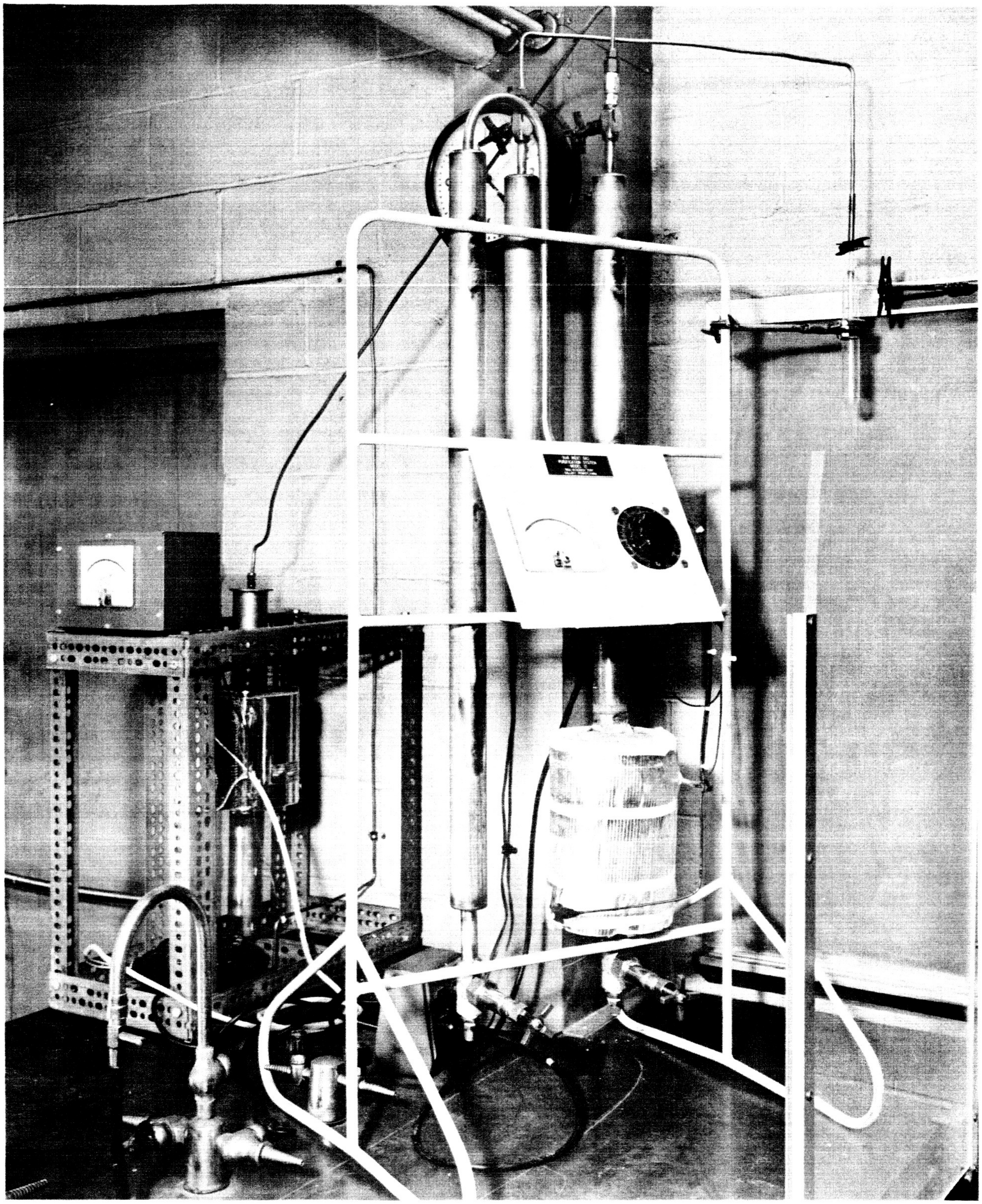

FIGURE 7 Helium Purification System - Titanium Sponge Furnace and NaK Bubbler

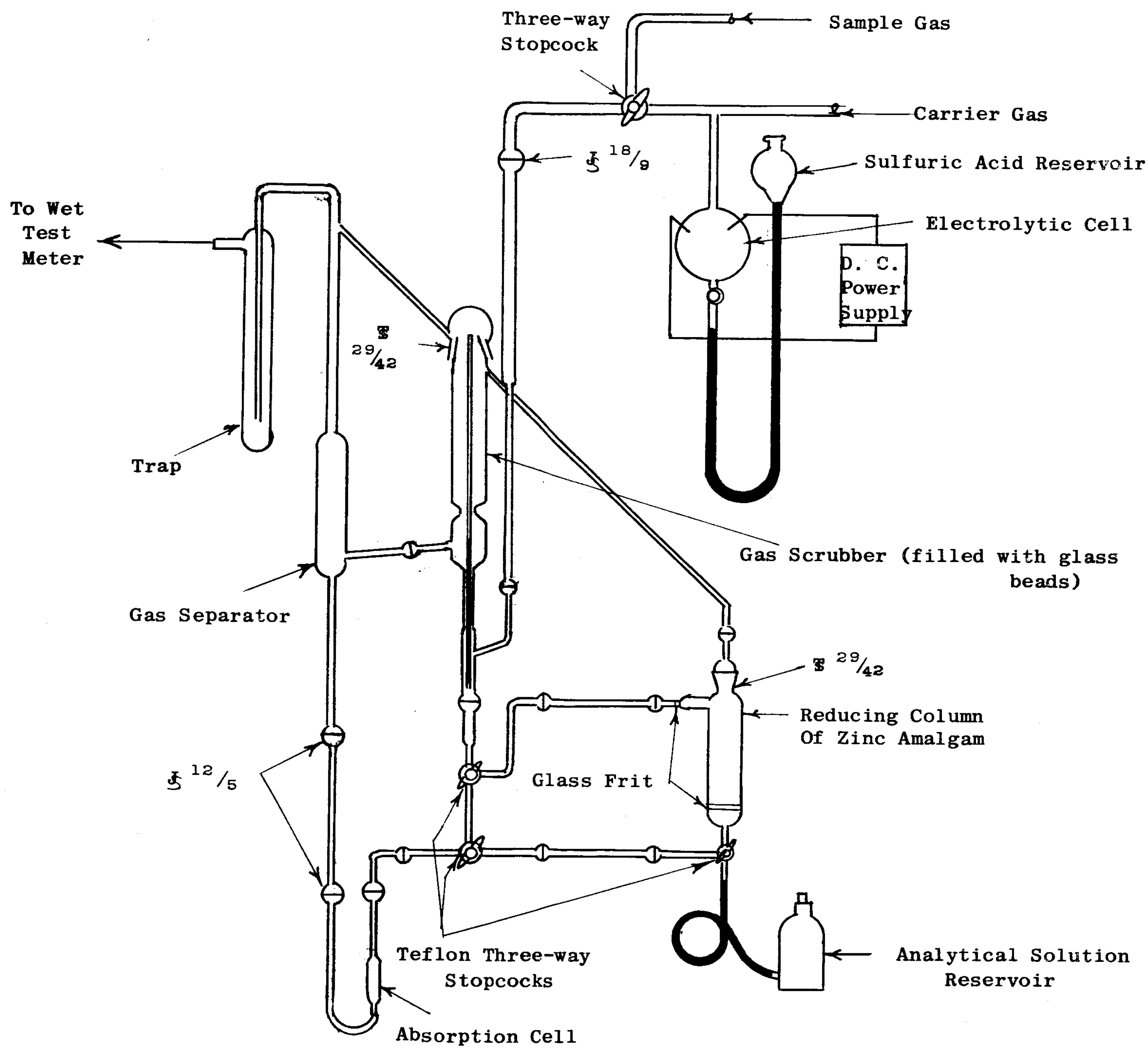

FIGURE 8 Diagram of Modified Brady Apparatus

colorless. The change in absorbance is observed on a spectrophotometer by flowing continuously the solution from a closed volume past a sensitive photocell. The calibration technique and data will appear in a future report.

b) An improved straight-through absorption cell with precision parallel glass sides (1 cm). This replaces the usual test tube-like cell wherein the mixing and flow of the reagent solution is slow and less responsive photometrically. Figure 9 shows the cell with two socket joints for interconnection with the remainder of the apparatus. The analytical portions of the glass system have been painted black.

c) A high precision narrow-band spectrophotometer in conjunction with a recorder for recording transmission changes versus time at a fixed wave-length. A new model #139 Hitachi Perkin-Elmer Spectrophotometer (shown with the Brady apparatus in Figure 10) will be used. The grating has 1440 lines/mm and a slit width adjustable from 0.005 to 3.0 mm. In conjunction with the Brady apparatus and the unidirectional cell, the calibration will be made to relate the total accumulative weight of oxygen (as opposed to the instantaneous amount) to elapsed time. Full scale will represent a 10% change in transmission as compared to 0-100% transmission formerly used. To simplify eventual read-out during an analytical run, the results will be monitored on the Varicord Model 43 linear-log recorder also shown in Figure 10. Adjustable ranges on both the Spectrophotometer and the Varicord will allow for the plotting of percent transmission versus elapsed time at constant wave length to obtain precise oxygen measurements. Changes in chart speed may be made to determine optimum reading conditions.

3. The over-all preliminary designed analytical system is shown in Figure 1. The bromine trifluoride will be transferred to the monel storage and purification vessel by prior evacuation of the vessel. Here the bromine trifluoride may be purified by exposure to vacuum, bubbling pure helium through the liquid, and freezing with liquid nitrogen. By means of helium pressure, the bromine trifluoride may be transferred to a Kel-F measuring tube and required amounts may then be transferred to the reaction vessel itself.

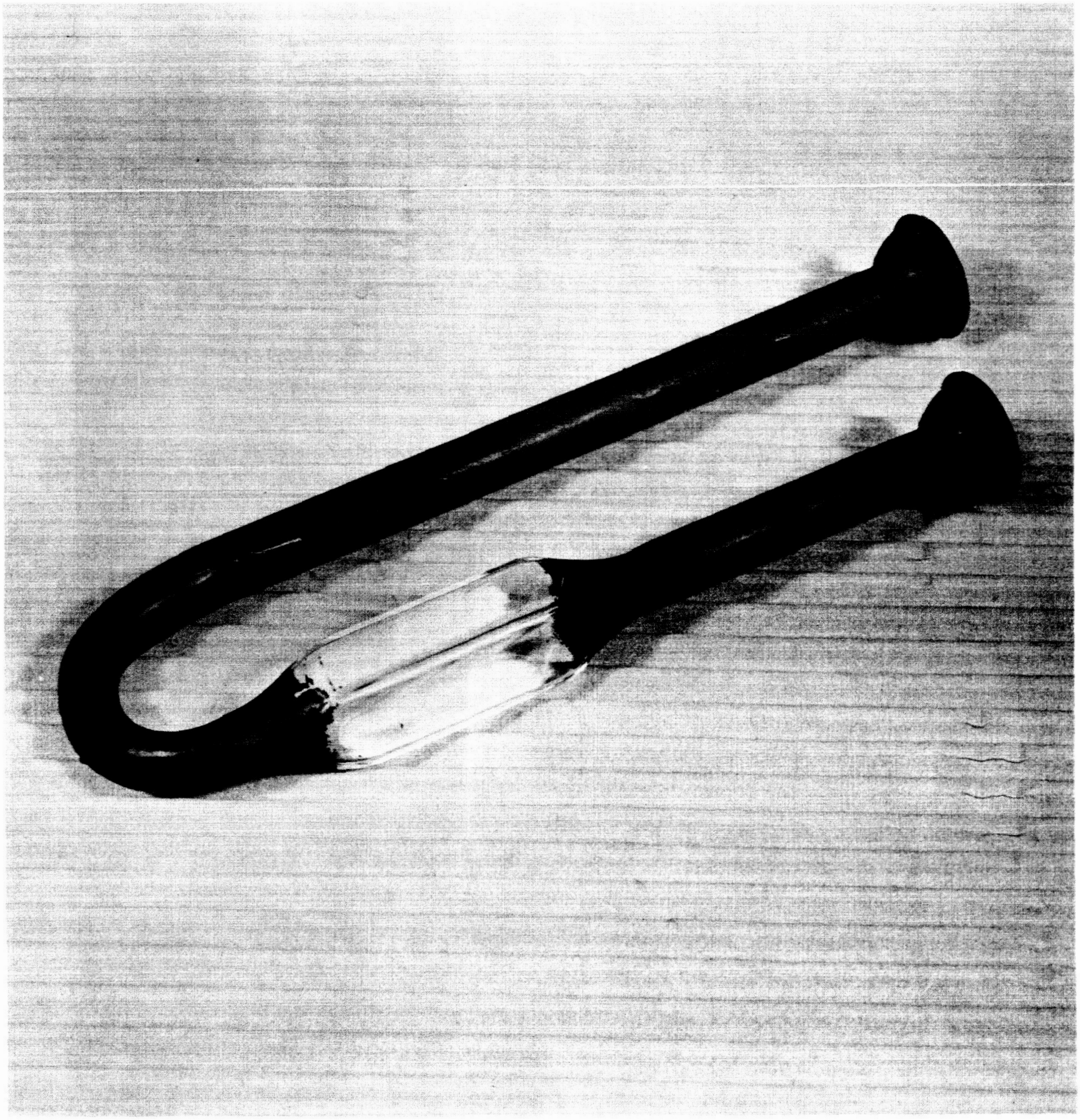

FIGURE 9 Absorption Cell for Brady Apparatus

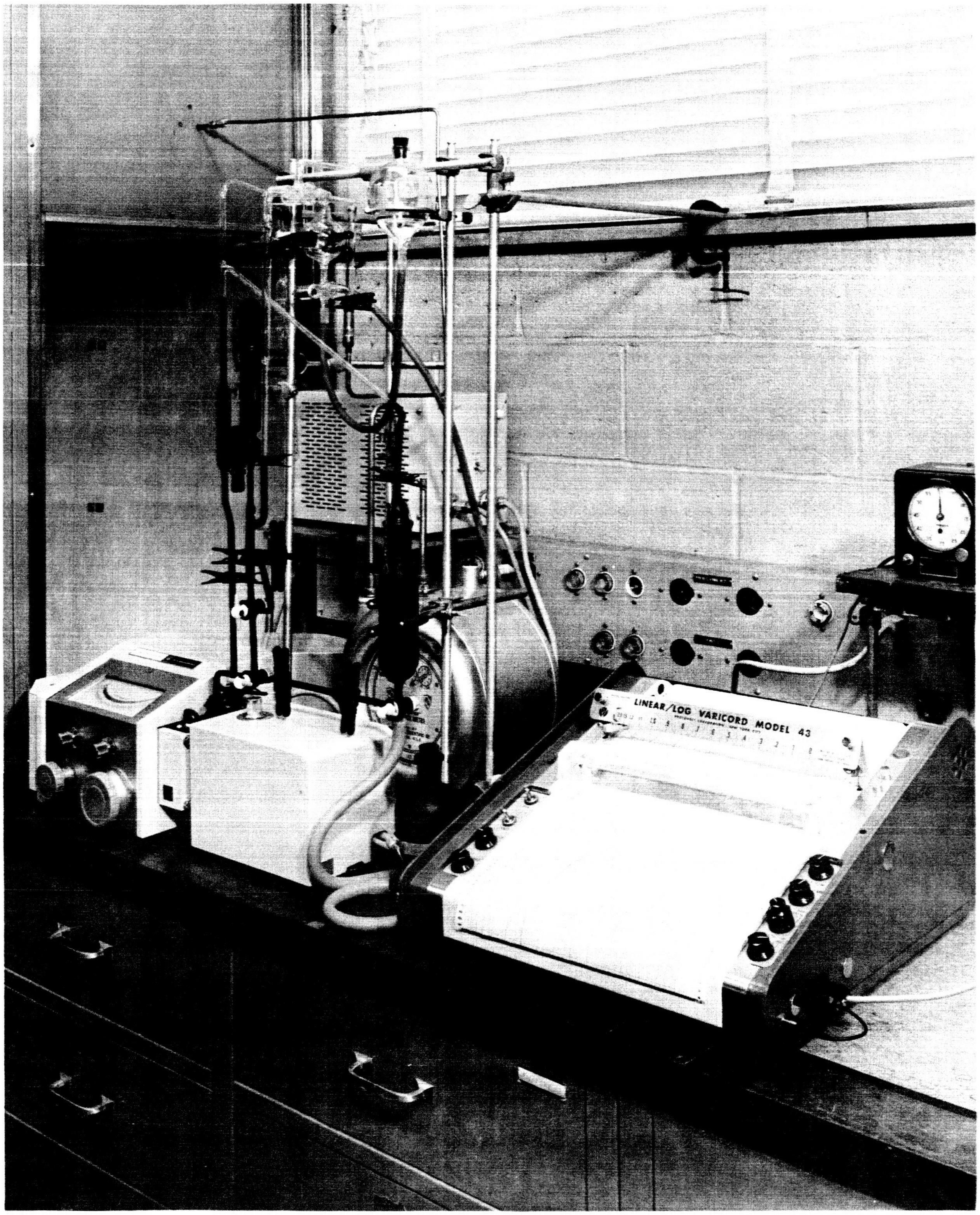

FIGURE 10 Oxygen Measuring System - Brady Apparatus and Perkin-Elmer Spectrophotometer

The monel adaptor (Figure 3) placed between the Hills McCanna ball valve and the reaction vessel, contains the necessary connecting parts for vacuum, helium supply, and communication to other portions of the apparatus. Following reaction with potassium, helium may be bubbled through the bromine trifluoride to remove oxygen. The gas will then pass through two monel cold traps containing a suitable refrigerant and thence to the Brady apparatus.

The reaction cell has been made large enough to run about 6-8 samples before disassembly. The feed-in system will allow for helium to flow both over the solidified BrF_3 or through the liquified BrF_3. In evacuating the reaction cell and the auxiliary communication lines, the main high vacuum system will be by-passed to avoid contamination. The traps between the cell and exit end towards the measuring section have been redesigned so as to avoid clogging by carried-over BrF_3 during the analytical runs. A minimum of fittings will be used. All heliarc welded construction will prevail to insure no leakage. All sensitive parts will be monel, copper, or Teflon.

The cold traps above consist of two concentric vessels (see Figure 4); the inner one containing a suitable refrigerant. A Teflon "O" ring provides a seal between the two members. Cajon ultra-high vacuum connections will be placed between the various vessels so that sections of the apparatus may be easily removed. These fittings are of monel and may be equipped with either Teflon or nickel gaskets.

Valves which are exposed to BrF_3 or its vapors are $\frac{1}{4}$" Durco plug-type valves manufactured by the Duriron Company of Dayton, Ohio. These valves are constructed of monel with a Teflon sleeve surrounding the plug which acts as a sealant. Valve parts are equipped with $\frac{1}{4}$" female pipe threads. Teflon tape is used as a sealant on all pipe thread connections.

WORK PLANNED FOR SECOND QUARTER

1. The Brady Apparatus - Spectrophotometric section will be calibrated. This involves the electrolytic generation of oxygen from water and its admittance into the system. Faraday's law is used here.

2. Final assembly of total system and completion of leak checking. This involves the intercommunication of the helium purification system, the extruder, the reaction cell section, the BrF_3 storage section, and finally the oxygen read-out section.

3. A task under this part of the program will be the determination of the weight of potassium extruded per full screw turn on the extruder. A reproducible value will obviate the necessity for the weight determination after reaction with the bromine trifluoride and, therefore, permit the analytical determination of several potassium samples in the same volume batch of the interhalogen without opening the reaction cell. For this test, the extruded potassium will be dropped into a vessel, covered with hexane, reacted with HCl, and finally weighed as KCl.

4. The entire system shall then be calibrated and checked out to establish and record the blank or leak rate when the bromine trifluoride is in place. The objective shall be to obtain a final reading of less than 10 micrograms of oxygen per hour under conditions normally used for an analytical run with potassium.

5. For the additional measurements of oxygen in potassium, the design of a statistical method is now underway. This will allow for the various parameters such as sample size, limit of oxygen weight, precision, etc.